



Antibacterial cotton functionalized with olive oil for developing medical textiles

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To promote the use of natural antimicrobial agents and to reduce the bad environmental impact caused due to synthetic agents, a novel finishing solution of olive oil along with ethanol has been developed and then applied on cotton fabric at different concentrations by pad-dry-cure method. The finished fabrics are characterized by SEM, FTIR & TGA methods. It is found that olive oil is successfully bonded on the cotton fabric. The antimicrobial property of finished fabric is evaluated by AATCC- 147 method. The treated fabrics show excellent resistance to both *E.coli* and *S.aureus* bacteria before and after washing. As the concentration of oil is increased, the antimicrobial efficiency is found to be higher. The whiteness index of treated fabrics is decreased, due to the formation of yellow patches on the fabric surface. Several physical properties, such as mass, tensile strength and wrinkle recovery angle have been analyzed. The results show that, there is an increase in mass and wrinkle recovery angle of finished the fabric. However, there is a reduction in the tensile strength of the cotton fabric. The above findings will promote the use of natural antimicrobial agents for the production of eco –friendly and sustainable antimicrobial textiles.

Keywords: Antibacterial activity, Cotton fabric, Fabric finishing, Medical textile, Olive oil, Physical properties

1 Introduction

Microorganisms are microscopic and found everywhere on the earth. Microorganisms may exist in single-celled or multi-celled. The growth of microorganisms on textile material is a common problem. Due to the presence of moisture and huge surface area, the growth of microbes takes place on textile material. This growth can be found on both natural and synthetic textiles, which leads to several effects like unpleasant odour, staining, deterioration, degradation, and pigmentation in the quality of textile material¹⁻³.

Natural fibres, especially cotton are highly affected by bacteria, due to the presence of cellulose, which acts as food for bacteria, whereas synthetic fibers are resistant to microorganisms due to their hydrophobic nature. To overcome these drawbacks, it is necessary to prevent the growth of microbes on textile material for providing good comfort and safety to the wearer.

The process of antimicrobial finishing imparts the ability of textile materials to inhibit the growth of microorganism, like *E.coli* and *S.aureus* on the surface of the material. The production of

antimicrobial textiles is carried out by the application of antimicrobial agents on the surface of textile material. These antimicrobial finished textiles protect both the users from pathogenic or odor generation microorganism and damage to the textile material. Recently, the demand for antimicrobial finished textiles has increased in medical, healthcare, surgical and sports activities to avoid cross-infection diseases⁴⁻⁶. These antimicrobial finished textiles have important functions, such as (i) protect the textile material from the damages caused due to microorganisms (ii) protect textile user against odor or pathogenic microorganisms and (iii) reduce odor and pigmentation^{7,8}.

In the last few decades, some research work has been carried out for the development of antimicrobial textiles by using synthetic antimicrobial agents. Synthetic antimicrobial agents are highly effective against bacteria. The textile materials treated with synthetic antimicrobial agents are durable and last for several washes, but they also lead to several disadvantages, and hence cause of water pollution, hazardous to human health and several other side effects⁹⁻¹¹. Synthetic agents, like silver nanoparticles, titanium nanoparticles, and other heavy metals, are non-biodegradable and hence affect the environment. These drawbacks limit the use of synthetic

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antimicrobial agents and created necessity for the development of eco-friendly antimicrobial finishes prepared from a natural resource like plants, essential oils, and other natural products.

The use of natural antimicrobial agents came into existence due to their effective antimicrobial activity. In the previous studies, several natural antimicrobial agents have been applied onto textiles, which showed excellent antimicrobial property. Essential oils extracted from various parts of plants, like flower, roots, stems, and leaves, possess great potential as antimicrobial agents^{12,13}. Several essential oils, like tea tree oil, patchouli oil, neem oil, eucalyptus oil, clove oil, rosemary oil, and, moxa oil, have been used in previous research for different functional properties, like antibacterial, fragrance and, aroma finish by different mode of finishing like direct application and microencapsulation method. Essential oil poses various advantages, like ecofriendly, sustainable textiles and, less side effect to human skin. On the other hand, they also show some disadvantages, like less durability, poor fastness properties, and, poor physical properties of textiles. Olive oil extracted from olive tree is the major product of olive fruit. Olive oil is mainly composed of triacylglycerols and fatty acids. Fatty acids are almost straight chain aliphatic carboxylic acids bonded with hydrogen atoms. One end of the terminal of fatty acid has a carboxylic group, which is reactive portion of the molecule and will take part in chemical reaction. Naturally occurring fatty acids share a common biosynthesis, and the chain is built from two carbon units. Especially *cis* double bonds are inserted by enzymes at appropriate positions related to carboxylic group which results in even chain length fatty acids with characteristic pattern of methylene interrupted *cis* double bonds. Generally, oils contain fatty acids with chain lengths between C₁₆ and C₂₂, with C₁₈ fatty acids, dominating in most plant oils like olive oil. Oleic acid, the major fatty acid which makes 55-83% of olive oil, is responsible for the antimicrobial property of olive oil. Oleic acid is typically monounsaturated fatty acid. Several research reports showed that the oil extracted from olive fruits and olive leaves showed excellent antimicrobial activity against bacteria¹⁴⁻¹⁶. Olive oil alongside with products obtained from olive tree has several unique medicinal, antibacterial, antioxidant and anti-inflammatory properties. The effect of olive oil on pain relief, wound healing, and cancer treatment has been proven. Moreover, in addition to sensory properties of olive

oil, addition of olive oil to daily diet can enhance the quality and safety of the food via antioxidant and antimicrobial compounds present in the oil. Phenolic compounds derived from olive oil has been proven to have significant anti-inflammatory properties. *In vitro* and *in vivo* research has suggested that the dietary intake of olive oil, containing significant concentrations of phenolic, may attenuate inflammatory responses in the body and therefore reduce the risk of chronic inflammatory disease development^{17,18}. To the best of our knowledge no report concerning the use of olive oil and its application on cotton fabric to produce an antimicrobial textile material has been published in the research.

Therefore, in this research work, olive oil has been applied on cotton fabric by the direct application at different concentrations. The antimicrobial property of treated cotton fabric is tested against *S.aureus* and *E.coli*. The durability of antimicrobial finishing before and after washing is also tested. Several physical properties, like mass, tensile strength, stiffness and wrinkle recovery angle of both treated and untreated samples, are tested. Moreover, FTIR, TGA, and SEM have been used for the characterization of treated and untreated cotton fabrics.

2 Materials and Methods

2.1 Materials

A plain weave 40s woven pure cotton fabric, supplied by Shandong Lufeng Co. LTD (110 EPIx85 PPI) weighing 124 g/m², was used in the study. Pure olive oil extracted from olive tree, supplied by Sinopharm Chemical Reagent CO. LTD, was used as an antimicrobial agent. Absolute ethanol, used as a solvent, was obtained from Sinopharm Chemical Reagent CO. LTD.

2.2 Methods

Preparation of Finishing Solution

Olive oil was used as an antimicrobial agent and ethanol was used as a solvent due to its excellent miscible nature with olive oil. The required amount of olive oil was added in ethanol and stirred for 5 min in a homogenizer for proper mixing of oil. Three different concentrations of solution (5 wt. %, 8 wt. % and 12 wt. %) were prepared.

Fabric Finishing

Cotton fabric was treated with different concentrations of olive oil ranging from 5, 8 & 12 wt. % by using a two-bowl horizontal laboratory paddler

(Xiamen Rapid CO. LTD) with pressure of 1.5 kg/cm² and speed of 2 RPM using a two-dip and two-nip process to give a wet pick up of 80%. After padding, the treated cotton samples were dried in an oven (Exin Instrument DGG-7070BD) at 60 °C for 5 min and then cured in a lab stenter (Xiamen Rapid CO. LTD) at 150 °C for 2 min.

Wash Fastness

Half of the olive oil-treated samples were washed in a Launder-O-meter (Hi-Tech Electronics Corp) at 40 °C for 45 min with M:L ratio of 1:30.

Antibacterial Activity

The antibacterial activity of the olive-oil treated (washed and unwashed) and untreated (blank) samples were evaluated qualitatively by using the AATCC 147-2011 method. Two different types of bacteria, viz *S.aureus* (gram-positive) and *E.coli* (gram-negative) were prepared to use in the test. The cell concentration of bacteria for this test was nutrient agar plates, which were streaked with five lines of bacteria from an inoculation loop. Then, all the samples (25 mm×50 mm) were placed over the incubated agar culture of the petri dishes. All the petri dishes were incubated at 37 °C for 18-24 h. After incubation for 18-24 h, the bacterial reduction was observed underneath as well as around the sample. The antibacterial activity was evaluated by the following equation:

$$W = (T-D)/2 \quad \dots (1)$$

where W is the width of the clear zone of inhibition in mm; T , the total diameter of the test specimen and clear zone in mm; and D , the diameter of the test specimen in mm.

FTIR Study

The FTIR of cotton samples and olive oil were measured using AVATAR 380 (Thermo Electron, USA). The wavenumber range was 500-4000 cm⁻¹ and the resolution was 1 cm⁻¹.

SEM Study

The surface morphology of treated, untreated and washed samples were obtained under SEM (HITACHI TM-1000) at a magnification of × 2.0k and × 5.0k using an acceleration voltage of 5KV. Before the assessment, all the samples were sputtered under vacuum with gold.

TG Analysis Study

TGA was performed on treated and untreated samples using TG 209 F1 Libra® (NETZSCH, Germany) analyzer to investigate the changes in

thermal stability by heating the finished and unfinished cotton samples till 800 °C with an increasing rate of 20 °C/min under Nitrogen steady flow.

Whiteness Measurement

The CIE whiteness index of all the fabrics was evaluated with a color measurement spectrophotometer (Datacolor 650, Datacolor, USA) tools. Each sample was folded twice and measured four times at different portions of the fabric surface and the average value was recorded.

Fabric Mass Study

To estimate the amount of olive oil attached to the fabric, the total mass of all the samples was determined by the ISO 3801 method.

Tensile Strength Study

Initially, the samples were conditioned in the standard atmosphere (25 °C, 65% RH) for 24h. The tensile strength measurements of fabric strip (20 cm × 5 cm) were made on Instron YG(B)033D (Darong Textile Instrument Co. LTD China) by using ASTM D5035-06 method in warp way.

Wrinkle Recovery Angle

Standard AATCC 66-2003 test method was used to measure the wrinkle recovery angle (Type-SDL, company-Dimerlo Express, Country-England) in both warp way and weft way. For each sample, 3 specimens were tested and the average value was reported

3 Results and Discussion

3.1 FTIR Study

Figure 1 shows the comparison of different FTIR spectra of pure olive oil and different cotton samples. Figure 1(a) represents the spectra for untreated cotton fabric. Figure 1(b) represents the spectra for washed sample after treatment. Figures 1(c)-(e) suggest the spectra for finished samples at different concentrations of the solution, such as 12 wt. %, 8 wt. % and 5 wt. % respectively. Figure 1(f) represents the spectra for peaks of pure olive oil. Comparison with spectra [Figs 1(b)-(e)] show that the shapes of the spectra of fabric are almost the same. From Figs 1(a)- (e), it can be seen that the cotton fabric has the adsorption peak of -OH at 3200 -3400 cm⁻¹. Figures 1(b)- (e) represent the FTIR absorption spectrum of the finished cotton, in which the peaks of triglyceride (the major functional group of olive oil) are formed at 2922 cm⁻¹ and 1743⁻¹ respectively along with the -OH group adsorption peak at 3332 cm⁻¹. There is no major difference observed between the adsorption peaks of finished and washed samples. Figure 1(f), shows the

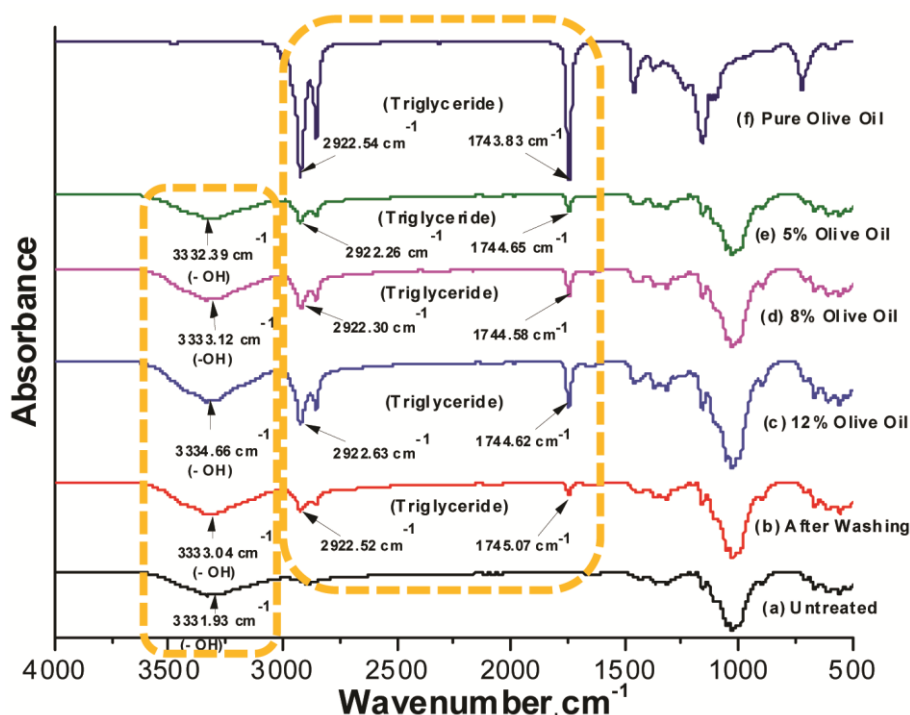


Fig. 1 — Infrared spectra of (a) untreated cotton, (b) washed cotton, (c) 12% olive oil treated cotton, (d) 8% olive oil treated cotton, (e) 5% olive oil treated cotton, and (f) pure olive oil

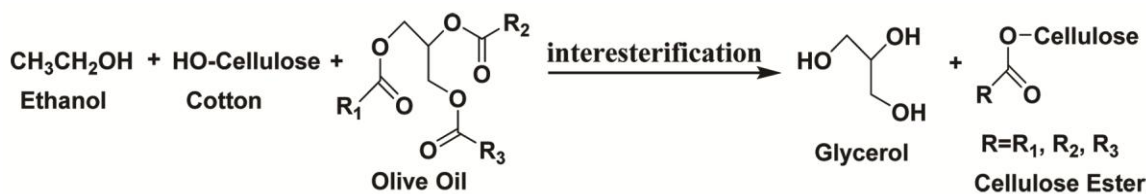


Fig. 2 — Possible reaction equation between olive oil and cotton fabric

presence of triglycerides at the adsorption peak of 2922 cm^{-1} and 1743 cm^{-1} . FTIR of untreated samples indicates the adsorption peaks of -OH group only [Fig. 1(a)]^{19,20}.

From the above analysis, it is found that the olive oil is successfully applied on cotton fabric. The presence of both functional groups of olive oil and cotton (triglyceride & OH) in FTIR spectra [Figs 1(b)-(e)] proved that, there is strong grafting of olive oil on cotton material. A chemical reaction occur between cotton fabric and olive oil at the conditions of catalyst and curing

A chemical reaction occur between the cotton fabric and the olive oil at the conditions of catalyst and curing, and the possible reaction equation is shown in Fig. 2.

3.2 Surface Morphology of Cotton Fabric

The surface morphology of olive oil finished, unfinished and washed cotton fabric is studied by scanning electron microscope.

Figure 3(a) shows the SEM photograph of untreated cotton fabric, wherein the surface is found to be smooth and clean with normal fibrils that are apparently found on the fabric surface. Figures 3(b)-(d) are the surface morphology of cotton fabric treated with 5 wt. %, 8 wt. % and, 12 wt. % olive oil respectively. The treated samples show uneven and rough surfaces with deposition of thick films, which confirms the adhesion of olive oil on the cotton fiber surface. The SEM images of washed cotton samples are shown in Fig. 4(e). It is noticed that a thin film of olive oil is partially discharged due to mechanical action during washing from the cotton fabric surface.

3.3 Thermal Properties

The TG curves of treated and untreated cotton fabrics under the nitrogen atmosphere are shown in the Fig. 4. The rate of weight reduction of the untreated sample is observed at about $330\text{-}370^\circ\text{C}$.

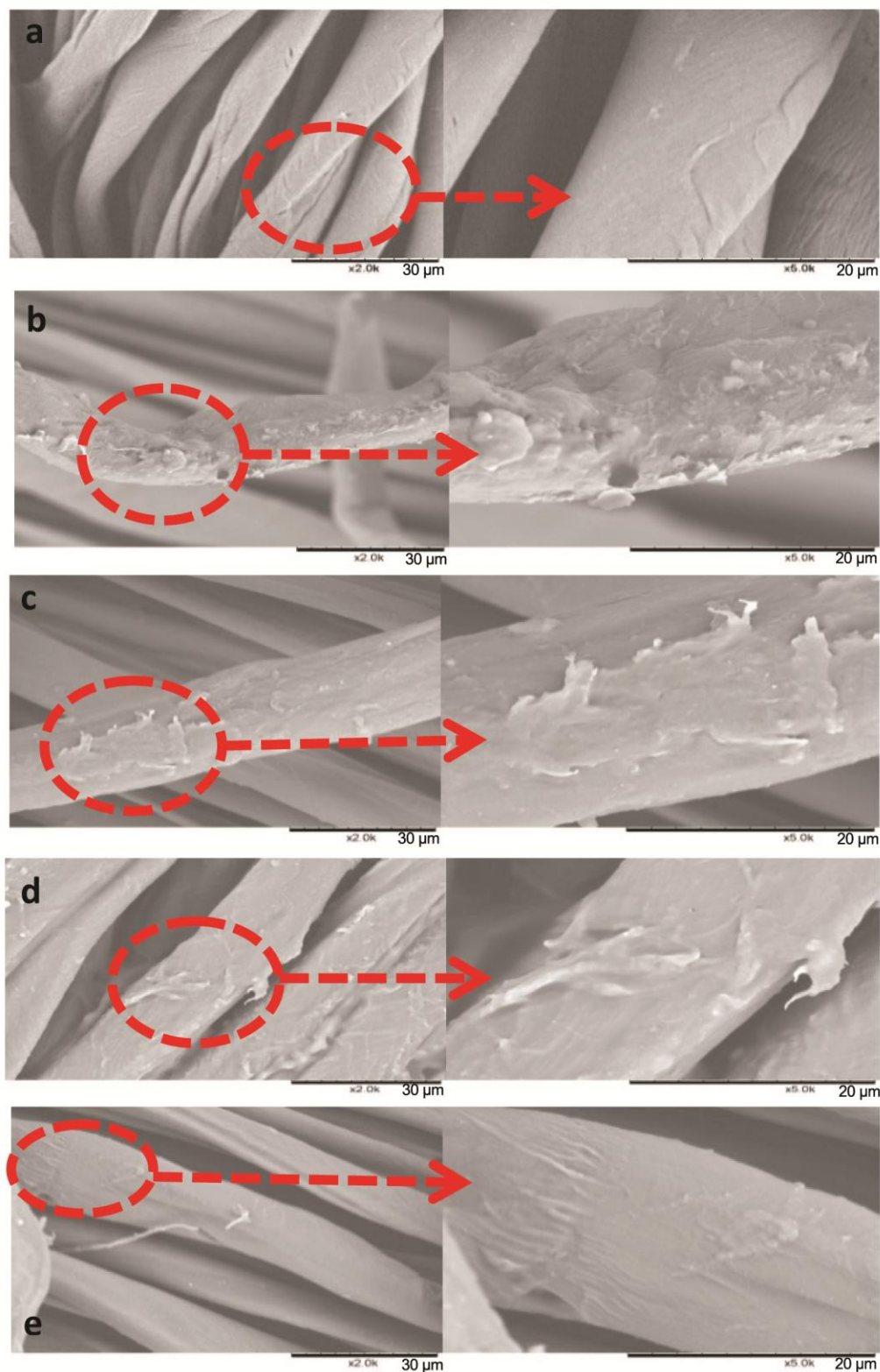


Fig. 3 — SEM photographs of cotton fabric (a) untreated cotton, (b) 5% olive oil treated cotton, (c) 8% olive oil treated cotton, (d) 12% olive oil treated cotton, and (e) washed cotton

Similarly, the weight reduction rate of the treated sample is found at 320-380 °C. The main degradation temperature for the treated sample is found 380 °C and that of untreated sample is 370 °C. However, from the above TG graph, it is concluded that the TG curve of treated cotton fabric is almost similar. There are no major differences in the weight reduction rate of both treated and untreated samples.

3.4 Antimicrobial Property

The finished, unfinished and washed samples have been assessed for antibacterial activity against G-negative (*E.coli*) and G-positive (*S.aureus*) using AATCC 147-2004 (Parallel streak method). The

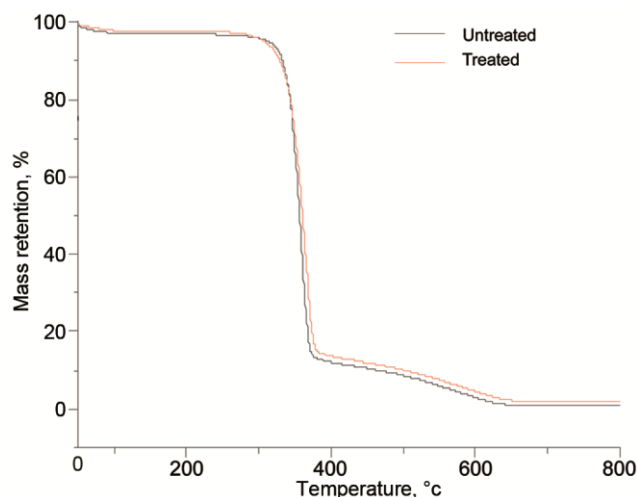


Fig. 4 — TGA curves of unfinished and finished cotton fabric by olive oil

results are shown in Fig. 5. It is found that there is bacterial growth of both *S.aureus* and *E.coli* for untreated samples [Figs 5 (a) & (b)]. There is no zone of inhibition formed for untreated samples. Figure 5(c) & (d) show a zone of inhibition up to a small extent of 5 wt.% sample. In Figs 5 (e) & (f), the zone of inhibition is comparatively higher than in 5 wt. % sample. In Figs 5(g) & (h), there is an excellent width of zone of inhibition for both *S.aureus* and *E.coli*, killing the maximum amount of bacteria.

Table 1 shows that in all the treated and washed samples, there is a good zone of inhibition ranging from 6 mm to 9 mm for *E.coli* and 12.5 mm to 16 mm for *S. aureus*. However, the width of the zone of inhibition for *S.aureus* is high in all different types of samples. Hence, it could be noted that the antibacterial efficacy of olive oil is found to be better against *S.aureus* than against *E.coli*. This may be due to the Gram-positive bacteria being more susceptible to olive oil extracts as compared to Gram-negative bacteria. As the concentration of olive oil is increased,

Table 1 — Antimicrobial test results

Fabric	Microbial growth	Zone of Inhibition, mm	
		<i>E.coli</i>	<i>S.aureus</i>
Untreated	Present	Nil	Nil
5% treated	Absent	7.5	12.5
8% treated	Absent	8	15.5
12% treated	Absent	9	16
Treated washed	Absent	6	15

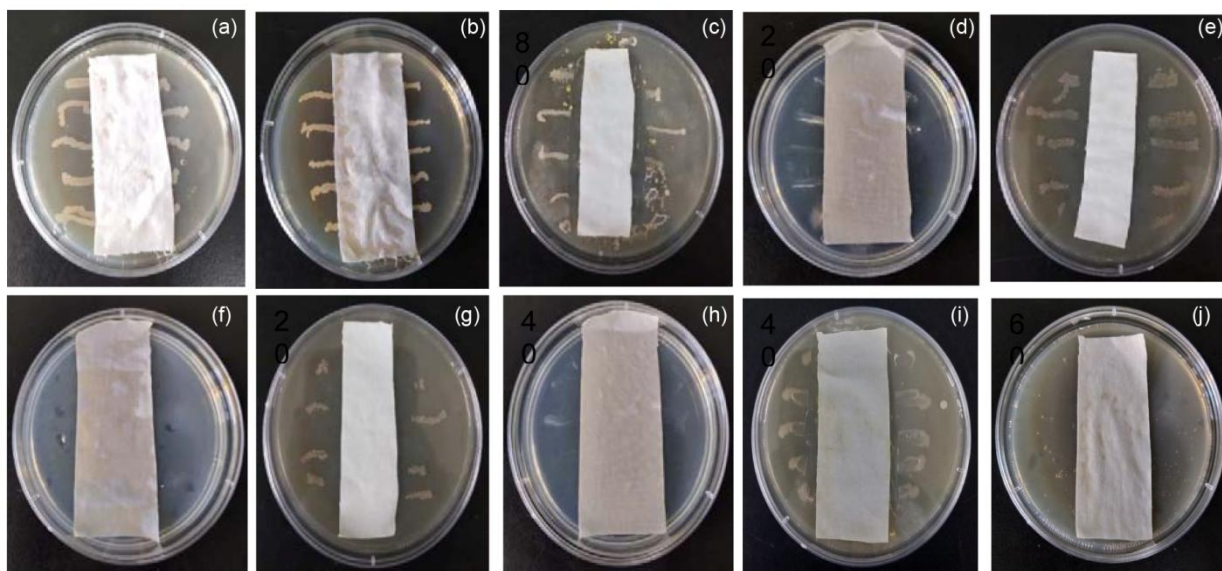


Fig. 5 — Antimicrobial activities of unfinished cotton fabric by (a) *E. coli* and (b) *S. aureus* as well as of finished cotton fabric with 5 wt. % olive oil by (c) *E. coli* and (d) *S. aureus*, with 8 wt. % olive oil by (e) *E.coli* and (f) *S.aureus*, with 12 wt. % olive oil (g) *E. coli* and (h) *S. aureus*, with 12 wt. % olive oil by (i) *E.coli* and (j) *S. Aureus*

the width of zone of inhibition is high. The highest width of the zone of inhibition is found at 12 wt. % concentration for both *E.coli* and *S.aureus*. The antimicrobial property of treated washed samples is found to be excellent, but there is a slight reduction in antibacterial efficacy. There is a slight reduction in the width of the zone of inhibition of the washed samples also. Hence, it could be concluded that olive-oil treated samples show excellent antimicrobial properties for both treated and washed against *E.coli* and *S.aureus* at all concentrations.

3.5 Whiteness Index

Figure 6 shows that the addition of olive oil reduces the whiteness index of fabric. As the concentration of olive oil is increased, the whiteness index is found to be decreased. The excessive amount of olive oil made the fabric yellow. These results verified that the presence of olive oil deposited on the cotton fibers. The maximum whiteness index is obtained for the treated washed samples; there is a 51% raise in the whiteness Index of washed samples and untreated samples.

3.6 Physical Properties

The properties of unfinished, finished and washed cotton fabrics such as mass, tensile strength, and wrinkle recovery angle have been investigated. From Table 2, it is found that the mass of fabric is increased as the concentration of olive oil increases. The fabric of the highest concentration (12 wt. %) shows the maximum mass. This is due to the presence of olive oil in the fabric. However, after washing the mass is reduced due to the loss of olive oil from the fabric. There is an increase in tensile strength of the finished fabric due to presence of oil and ethanol in the fibres, which creates strong bonds in the fiber morphology. Less force is required to break the untreated cotton fabric, whereas more force is required for untreated cotton fabric. Also, the wrinkle recovery angle of both warp and weft is mentioned in the table. The WRA is increased as the concentration of olive oil is increased. This is due to the deposition of chemicals in pores of cotton fabric, which creates strong bonds in the cotton fabric.

3.7 Aesthetic Appearance of Fabrics

After finishing the samples, the whiteness index of the samples is found to be reduced. Yellow patches are formed on the fabric surface, giving a strong smell of olive oil due to presence of finishing solution. The feel of cotton fabric is found to be harsh and rough due to curing the fabric at 150°C.

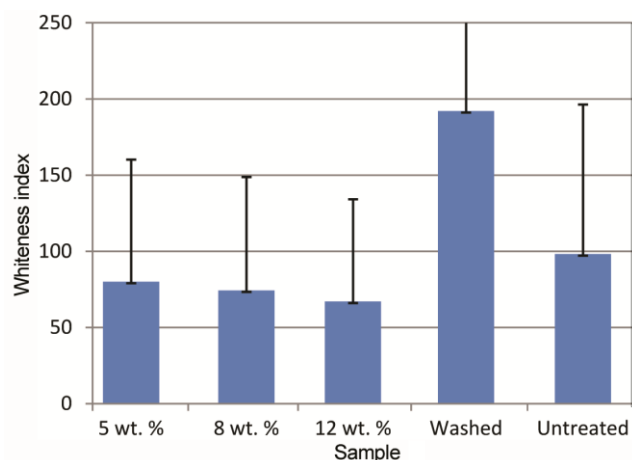


Fig. 6 — Whiteness index of untreated and treated samples

Table 2 — Physical properties of untreated and finished fabric

Cotton	Avg. wrinkle recovery angle (W+F), deg	Avg. tensile strength N	Mass, g/m ²
Untreated cotton	76	+00232.5	124.52
5 wt. % treated cotton	82	+00333.2	128.53
8 wt. % treated cotton	95	+00339.8	130.07
12 wt. % treated cotton	105	+00351.1	135.41
Washed cotton	80	+00275.2	127.11
Standard deviation	10.78	45.31	3.63
CV %	12.30	14.79	2.82

4 Conclusion

In this work, olive oil is successfully grafted on cellulose material by the pad-dry-cure method of finishing. The results of FTIR and SEM images show the presence and grafting of olive oil on cotton fabric. However, there is no major difference in between the thermal properties of treated and untreated cotton fabric. The treated cotton fabrics show better antimicrobial properties even after washing. Hence, the antimicrobial property is concentration-dependent for which 12 wt. % is the optimized concentration of olive oil in this research. The treatment decreases the whiteness index of treated fabric, whereas the mass and wrinkle recovery angle is increased, as there is an increase in the concentration of olive oil. Hence, pad-dry-cure is a desirable method for finishing textiles. This study paved the way for natural antimicrobial agents. Several essential oils like olive oil can be used as a grafting material to achieve an antimicrobial activity in a textile material for biomedical application. However, further research work can be carried out by increasing the concentration of olive oil to get maximum antimicrobial efficacy with improved aesthetic appearance.

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